

N-(3,4-Dichlorophenyl)methanesulfonamide

 B. Thimme Gowda,^{a*} Sabine Foro^b and Hartmut Fuess^b

^aDepartment of Chemistry, Mangalore University, Mangalagangothri 574 199, Mangalore, India, and ^bInstitute of Materials Science, Darmstadt University of Technology, Petersenstrasse 23, D-64287 Darmstadt, Germany

Correspondence e-mail: gowdabt@yahoo.com

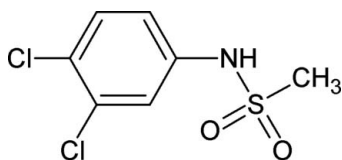
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Key indicators: single-crystal X-ray study; $T = 299$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.034; wR factor = 0.094; data-to-parameter ratio = 13.7.

In the structure of the title compound, $\text{C}_7\text{H}_7\text{Cl}_2\text{NO}_2\text{S}$, the conformation of the N—H bond is *syn* to the *meta*-chloro group, in contrast with the *anti* conformation observed for the *meta*-methyl group in *N*-(3,4-dimethylphenyl)methanesulfonamide and the conformation lying between *syn* and *anti* to the *meta*-chloro substituent in *N*-(3-chlorophenyl)methanesulfonamide. The bond parameters are similar to those in other methanesulfonamides, except for some differences in the bond and torsion angles. There are N—H···O hydrogen bonds, resulting in the formation of dimers. C—H···O interactions between the dimers lead to a ribbon-like structure.

Related literature

For related structures, see: Gowda *et al.* (2007*a,b,c,d,e,f*); Klug (1968). For the synthetic procedure, see: Jayalakshmi & Gowda (2004).



Experimental

Crystal data

$\text{C}_7\text{H}_7\text{Cl}_2\text{NO}_2\text{S}$
 $M_r = 240.10$
 Triclinic, $P\bar{1}$
 $a = 5.192$ (1) Å
 $b = 8.452$ (1) Å
 $c = 11.031$ (1) Å
 $\alpha = 79.72$ (1)°
 $\beta = 89.72$ (1)°

$\gamma = 84.89$ (1)°
 $V = 474.38$ (12) Å³
 $Z = 2$
 Cu $K\alpha$ radiation
 $\mu = 7.95$ mm⁻¹
 $T = 299$ (2) K
 0.40 × 0.15 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.328$, $T_{\max} = 0.496$
 (expected range = 0.298–0.451)
 1916 measured reflections

1685 independent reflections
 1536 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.015$
 3 standard reflections
 frequency: 120 min
 intensity decay: 1.0%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.094$
 $S = 1.08$
 1685 reflections
 123 parameters
 1 restraint

H atoms treated by a mixture of independent and constrained refinement

$\Delta\rho_{\text{max}} = 0.27$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.33$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N5}-\text{H5}\cdots\text{O3}^{\text{i}}$	0.844 (10)	2.079 (12)	2.913 (3)	169 (3)
$\text{C10}-\text{H10}\cdots\text{O4}^{\text{ii}}$	0.93	2.60	3.352 (3)	138

Symmetry codes: (i) $-x, -y + 1, -z$; (ii) $-x, -y + 1, -z + 1$.

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1996); cell refinement: *CAD-4-PC Software*; data reduction: *REDU4* (Stoe, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2184).

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supplementary materials

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***N*-(3,4-Dichlorophenyl)methanesulfonamide**

B. T. Gowda, S. Foro and H. Fues

Comment

The structural studies of sulphonanilides are of interest due to their biological activity as the latter is thought to be due to the amide hydrogen portion of the molecules. In the present work, the structure of *N*-(3,4-dichlorophenyl)-methanesulfonamide (34DCPMSA) has been determined as part of our study of the substituent effects on the solid state structures of methanesulfonanilides (Gowda *et al.*, 2007*a, b, c, d, e, f*). The structure of 34DCPMSA (Fig. 1) is similar to those of other methanesulfonanilides (Gowda *et al.*, 2007*a, b, c, d, e, f*). The conformation of the N—H bond in 34DCPMSA is *syn* to the *meta* chloro group in contrast to the *anti* conformation observed for the *meta* methyl group in *N*-(3,4-dimethylphenyl)-methanesulfonamide (34DMPMSA) (Gowda *et al.*, 2007*c*) and the conformation lying between *syn* and *anti* to the *meta*-chloro substituent in *N*-(3-chlorophenyl)-methanesulfonamide (3CPMSA) (Gowda *et al.*, 2007*b*). The substitution of Cl atom at the *meta* position of *N*-(phenyl)-methanesulfonamide (PMSA) to produce 3CPMSA changes its space group from monoclinic $P2_1/c$ (Klug, 1968) to $C2/c$ (Gowda *et al.*, 2007*b*). The substitution of an additional chloro group at the *para* position of 3CPMSA to produce 34DCPMSA changes the space group from monoclinic $C2/c$ to triclinic $P-1$ in contrast to change over from orthorhombic $Pccn$ to monoclinic $P2_1$ on substitution of an additional methyl group at the *para* position in *N*-(3-methylphenyl)-methanesulfonamide to produce 34DMPMSA (Gowda *et al.*, 2007*c*). The bond parameters in 34DCPMSA are similar to those in PMSA (Klug, 1968), 3CPMSA (Gowda *et al.*, 2007*b*), 34DMPMSA (Gowda *et al.*, 2007*c*) and other methanesulfonanilides (Gowda *et al.*, 2007*a, d, e, f*), except for some difference in the bond and torsional angles. The amide H atom is nearly in the plane of the dichlorophenyl group, the N atom is 0.110 (3) Å above the plane whereas the S atom is -0.426 (2) Å below the plane. The methyl group is clearly below the plane. The N—H...O hydrogen bonds (Table 1) result in the formation of dimers. The C—H...O interactions among the dimers in turn develop into ribbon like structures (Fig. 2).

Experimental

The title compound was prepared according to the literature method (Jayalakshmi & Gowda, 2004). The purity of the compound was checked by determining its melting point. It was characterized by recording its infrared and NMR spectra (Jayalakshmi & Gowda, 2004). Single crystals of the title compound were obtained from a slow evaporation of its ethanolic solution and used for X-ray diffraction studied at room temperature.

Refinement

H atoms attached to C atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic) or 0.96 Å (CH₃) and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{aromatic})$ or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{CH}_3)$. H atom attached to N was refined using a N—H restraint of 0.85 (1) Å.

Figures

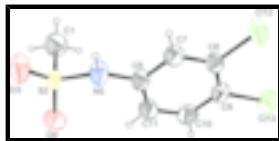


Fig. 1. Molecular structure of the title compound showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

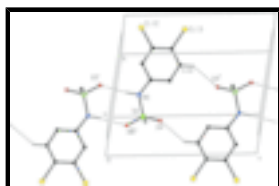


Fig. 2. Partial packing view showing the formation of the ribbon through N—H...O and C—H...O hydrogen bonding interactions. H bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry code: (i) $-x, -y + 1, -z$; (ii) $-x, -y + 1, -z + 1$].

N-(3,4-Dichlorophenyl)methanesulfonamide

Crystal data

$C_7H_7Cl_2NO_2S$

$M_r = 240.10$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 5.192\ (1)\ \text{\AA}$

$b = 8.452\ (1)\ \text{\AA}$

$c = 11.031\ (1)\ \text{\AA}$

$\alpha = 79.72\ (1)^\circ$

$\beta = 89.72\ (1)^\circ$

$\gamma = 84.89\ (1)^\circ$

$V = 474.38\ (12)\ \text{\AA}^3$

$Z = 2$

$F_{000} = 244$

$D_x = 1.681\ \text{Mg m}^{-3}$

Cu $K\alpha$ radiation

$\lambda = 1.54180\ \text{\AA}$

Cell parameters from 25 reflections

$\theta = 5.3\text{--}27.1^\circ$

$\mu = 7.95\ \text{mm}^{-1}$

$T = 299\ (2)\ \text{K}$

Prism, colourless

$0.40 \times 0.15 \times 0.10\ \text{mm}$

Data collection

Enraf–Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 299\ (2)\ \text{K}$

$\omega/2\theta$ scans

Absorption correction: ψ scan
(North *et al.*, 1968)

$T_{\min} = 0.328, T_{\max} = 0.496$

1916 measured reflections

1685 independent reflections

1536 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.015$

$\theta_{\max} = 66.9^\circ$

$\theta_{\min} = 4.1^\circ$

$h = -6 \rightarrow 1$

$k = -10 \rightarrow 10$

$l = -13 \rightarrow 13$

3 standard reflections

every 120 min

intensity decay: 1.0%

Refinement

Refinement on F^2

Secondary atom site location: difference Fourier map

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.034$$

$$wR(F^2) = 0.094$$

$$S = 1.08$$

1685 reflections

123 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0543P)^2 + 0.2324P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.27 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.33 \text{ e } \text{\AA}^{-3}$$

Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.2536 (6)	0.2368 (3)	0.2322 (3)	0.0596 (7)
H1A	0.3692	0.2599	0.2931	0.089*
H1B	0.3441	0.2355	0.1562	0.089*
H1C	0.1904	0.1333	0.2601	0.089*
C6	0.2776 (4)	0.6371 (3)	0.2260 (2)	0.0371 (5)
C7	0.4409 (5)	0.7364 (3)	0.1567 (2)	0.0388 (5)
H7	0.4471	0.7416	0.0718	0.047*
C8	0.5949 (4)	0.8279 (3)	0.2121 (2)	0.0378 (5)
C9	0.5897 (5)	0.8187 (3)	0.3392 (2)	0.0413 (5)
C10	0.4287 (5)	0.7178 (3)	0.4082 (2)	0.0465 (6)
H10	0.4258	0.7106	0.4933	0.056*
C11	0.2715 (5)	0.6270 (3)	0.3533 (2)	0.0443 (5)
H11	0.1626	0.5598	0.4008	0.053*
N5	0.1139 (4)	0.5562 (2)	0.16041 (18)	0.0456 (5)
O3	-0.1591 (4)	0.3621 (2)	0.10627 (16)	0.0565 (5)
O4	-0.1291 (4)	0.3894 (2)	0.32363 (17)	0.0547 (5)
S2	-0.00703 (11)	0.38570 (6)	0.20856 (5)	0.03832 (18)
Cl12	0.79029 (13)	0.95435 (8)	0.12089 (6)	0.0547 (2)
Cl13	0.77694 (14)	0.93584 (9)	0.41041 (7)	0.0603 (2)
H5	0.105 (6)	0.581 (3)	0.0828 (10)	0.054 (8)*

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Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0588 (16)	0.0464 (14)	0.0720 (19)	-0.0034 (12)	0.0038 (14)	-0.0075 (13)
C6	0.0464 (12)	0.0306 (10)	0.0340 (11)	-0.0072 (9)	-0.0007 (9)	-0.0028 (8)
C7	0.0507 (13)	0.0343 (11)	0.0302 (11)	-0.0062 (9)	0.0016 (9)	-0.0010 (9)
C8	0.0433 (12)	0.0302 (10)	0.0390 (11)	-0.0074 (9)	0.0024 (9)	-0.0018 (9)
C9	0.0465 (12)	0.0375 (12)	0.0422 (13)	-0.0059 (10)	-0.0019 (10)	-0.0125 (10)
C10	0.0589 (15)	0.0512 (14)	0.0311 (11)	-0.0096 (11)	0.0036 (10)	-0.0094 (10)
C11	0.0553 (14)	0.0451 (13)	0.0332 (12)	-0.0159 (11)	0.0087 (10)	-0.0036 (10)
N5	0.0635 (13)	0.0435 (11)	0.0301 (10)	-0.0233 (9)	-0.0029 (9)	0.0020 (8)
O3	0.0682 (12)	0.0577 (11)	0.0448 (10)	-0.0329 (9)	-0.0079 (8)	0.0010 (8)
O4	0.0618 (11)	0.0587 (11)	0.0464 (10)	-0.0213 (9)	0.0191 (8)	-0.0095 (8)
S2	0.0442 (3)	0.0374 (3)	0.0335 (3)	-0.0145 (2)	0.0033 (2)	-0.0018 (2)
Cl12	0.0603 (4)	0.0482 (4)	0.0546 (4)	-0.0244 (3)	0.0056 (3)	0.0028 (3)
Cl13	0.0638 (4)	0.0656 (4)	0.0600 (4)	-0.0216 (3)	-0.0032 (3)	-0.0260 (3)

Geometric parameters (\AA , $^\circ$)

C1—S2	1.751 (3)	C8—Cl12	1.728 (2)
C1—H1A	0.9600	C9—C10	1.377 (4)
C1—H1B	0.9600	C9—Cl13	1.732 (2)
C1—H1C	0.9600	C10—C11	1.380 (3)
C6—C7	1.378 (3)	C10—H10	0.9300
C6—C11	1.392 (3)	C11—H11	0.9300
C6—N5	1.414 (3)	N5—S2	1.624 (2)
C7—C8	1.376 (3)	N5—H5	0.844 (10)
C7—H7	0.9300	O3—S2	1.4329 (18)
C8—C9	1.390 (3)	O4—S2	1.4201 (18)
S2—C1—H1A	109.5	C8—C9—Cl13	120.81 (18)
S2—C1—H1B	109.5	C9—C10—C11	121.1 (2)
H1A—C1—H1B	109.5	C9—C10—H10	119.4
S2—C1—H1C	109.5	C11—C10—H10	119.4
H1A—C1—H1C	109.5	C10—C11—C6	119.3 (2)
H1B—C1—H1C	109.5	C10—C11—H11	120.4
C7—C6—C11	119.8 (2)	C6—C11—H11	120.4
C7—C6—N5	116.56 (19)	C6—N5—S2	127.66 (16)
C11—C6—N5	123.6 (2)	C6—N5—H5	119 (2)
C8—C7—C6	120.5 (2)	S2—N5—H5	111 (2)
C8—C7—H7	119.7	O4—S2—O3	118.85 (12)
C6—C7—H7	119.7	O4—S2—N5	109.71 (11)
C7—C8—C9	120.1 (2)	O3—S2—N5	104.15 (10)
C7—C8—Cl12	118.80 (17)	O4—S2—C1	107.99 (14)
C9—C8—Cl12	121.13 (18)	O3—S2—C1	108.85 (14)
C10—C9—C8	119.2 (2)	N5—S2—C1	106.67 (13)
C10—C9—Cl13	119.97 (18)		
C7—C6—N5—S2	152.95 (19)	C11—C6—N5—S2	-30.3 (4)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N5—H5···O3 ⁱ	0.844 (10)	2.079 (12)	2.913 (3)	169 (3)
C10—H10···O4 ⁱⁱ	0.93	2.60	3.352 (3)	138

Symmetry codes: (i) $-x, -y+1, -z$; (ii) $-x, -y+1, -z+1$.

Fig. 1

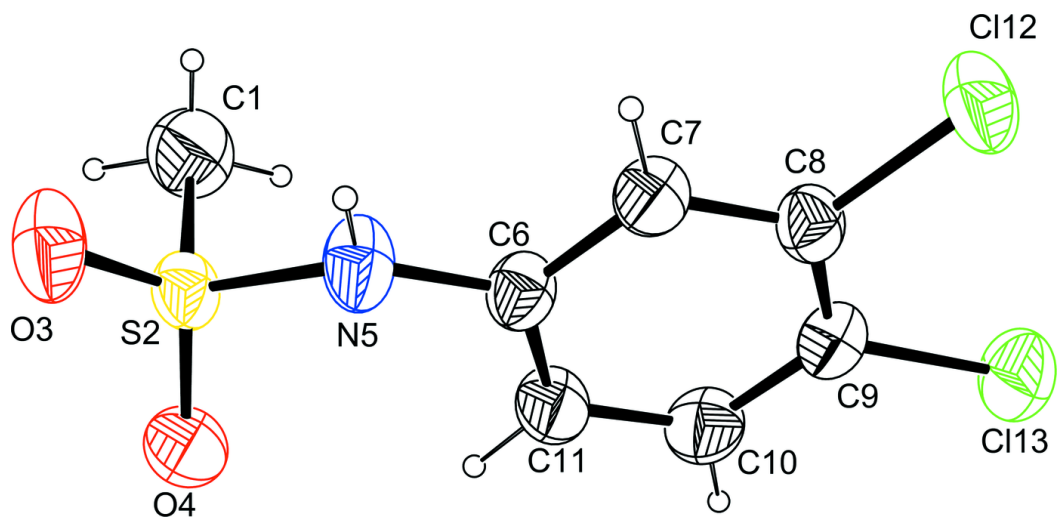


Fig. 2

